

Small amplitude Dynamic AFM: quantifying interactions with different tip detection and excitation schemes in presence of additional resonances.

Luca Costa¹, Mario S Rodrigues²

1- European Synchrotron Radiation Facility, 6 rue Jules Horowitz BP 220, 38043 Grenoble Cedex, France

2- CFMC/Dep. de Física, Universidade de Lisboa, Campo Grande 1749-016 Lisboa, Portugal

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Abstract

Quantifying the tip-sample interaction at the nanoscale in Amplitude Modulation mode AFM is challenging, especially when measuring in liquids. Here, we derive formulas for the tip-sample conservative and dissipative interactions and investigate the effect that spurious resonances have on the measured interaction. Both direct and acoustic excitation are considered. We also highlight the differences between measuring directly the tip position or the cantilever deflection. We show that, when probing the tip-sample forces, the acoustically excited cantilever behavior is insensitive to spurious resonances as long as the measured signal corresponds to the tip position, or if the excitation force is correctly taken into account. Since the effective excitation force may depend on the presence of such spurious resonances, we consider the cases where the frequency is kept constant during the measurement so that the proportionality between excitation signal and actual excitation force is kept constant. With the present work we show the advantages that result from the use of a calibration method based on the acquisition of approach-retract curves. Optical beam deflection based AFMs benefit from the use of this calibration method, especially in presence of spurious resonances in the cantilever transfer function.

1 Introduction

Dynamic AFM was introduced in the late 80s [1] as the natural evolution of the first Atomic Force Microscopes [2]. Thanks to its flexibility, Amplitude Modulation AFM (AM-AFM) [3] has become a successful dynamic operational scheme widely employed to characterize surfaces at the nanoscale. It has continuously evolved in terms of achievable lateral resolution and scan speed, producing impressive results both at the solid/gas interfaces in ambient conditions [4] and at the solid/liquid interfaces [5]. A complete overview is given by references [3] and [6]. In AM-AFM, micro-sized cantilevers are convention-

ally excited at a frequency close to their first eigenmode. The oscillation amplitude of the tip is the feedback signal kept constant to obtain the sample morphology during the scan. Recently, the technique evolved in large amount of operational schemes based on the simultaneous detection of several cantilever eigenmodes or harmonics of the tip displacement [7, 8].

Evidently, the cantilever excitation plays a central role in AM-AFM. Conventionally, a mechanical vibration in the cantilever holder is provided through the excitation of a small piezoelectric element (dither). The setup is widely employed in many commercial and custom-made AFMs and permits measurements both in air and in liquids. Despite the success of this method, the cantilever transfer function presents a forest of spurious peaks particularly when measuring in liquids. As a consequence, a quantitative estimation of the conservative and dissipative interaction between the probe and the sample is complicated. Moreover, it has already been observed that the motion of the cantilever base due to the acoustic excitation is not negligible in situations where the Q-factor is low, a typical situation when measuring in liquids. The same holds if the cantilever is not operated at a frequency close to its resonance frequency. Several solutions have been proposed to overcome the presence of spurious peaks in the cantilever transfer function. One possibility is the development of custom-made liquid cells which limit the presence of spurious excitations [9]. Another possibility is the direct excitation of the tip bypassing the conventional piezoelectric excitation. In particular, in the last decade scientists have introduced magnetic [10, 11], capacitive [12] and photothermal [13, 14] actuation schemes.

The differences between the direct excitation of the tip and the conventional dither excitation have already been studied and reported [15, 16]. Consistent efforts have been done to properly quantify the conservative and dissipative interactions when using acoustically excited cantilevers in liquids [17, 18]. The proposed methods have been successfully employed for AFMs based on an optical beam deflections scheme which provides a measurement of the tip bending angle [19].

Here we report a general study of the dynamics of acoustically excited cantilevers and lay-down formulas for deriv-

* Corresponding author: *mmrodrigues@fc.ul.pt*

ing the conservative and dissipative interactions considering different types of detection methods.

This results in a direct estimation of the conservative and dissipative interactions between the AFM probe and the specimen when employing Fabry-Perot interferometers as detection method [20, 21]. A second issue that is addressed in the present work is the consistent advantages that result from the use of approach-retracts curves as calibration method, as done with the Force Feedback Microscope[22, 23, 24], versus the standard characterization of the cantilever transfer function. With the described procedure, it would be possible to ignore the effects of spurious resonances when using optical beam deflection based AFMs.

Firstly, we derive simple equations to quantify the conservative and dissipative interactions in the small oscillation amplitudes regime assuming to know the tip position, which is the case of AFMs employing fiber-optic based interferometer. Then, we focus on the problem of the cantilever motion coupled with an additional oscillator which may be represented by a resonance of the liquid cell. Both cases where the cantilever is coupled to an extra oscillator at its base or directly at the tip position are considered. Finally, we report a description of the cantilever dynamics when the tip bending angle is measured, as in conventional optical beam deflection operational scheme. The calibration procedure based on the acquisition of approach-retract curves and the derived formulas have been used to characterize a tip-sample electrostatic interaction, even in presence of spurious resonances in the cantilever transfer function.

2 General formula for small oscillation amplitudes

In this section we will produce two general formulas for the interaction stiffness k_i and damping γ_i without assuming the whole system has a specific transfer function dominated by the cantilever transfer function. It will be assumed only that all the forces that are present are additive.

Consider a point mass which is being acted by two forces. One force given by $F_y(t)$ and another by $F_x(t)$. The two forces add together to make a resultant force $F_r(t)$, which determines the motion of the mass:

$$x(t) = A \cos(\omega t + \phi) \quad (1)$$

Which from Newton's second law means:

$$F_r = -mA\omega^2 \cos(\omega t + \phi) \quad (2)$$

If we are interested to know $F_y(t)$, in the case where the forces are additive, we can put $F_y(t) = F_r(t) - F_x(t)$ i.e.:

$$F_y(t) = A_r \cos(\omega t + \phi) - A_x \cos(\omega t) \quad (3)$$

Where A is the amplitude of the force identified with its respective subscript. F_x can be rewritten as:

$$F_x(t) = A_x[\cos(\omega t + \phi) \cos(\phi) + \sin(\omega t + \phi) \sin(\phi)] \quad (4)$$

From which we conclude:

$$F_y(t) = [A_r - A_x \cos(\phi)] \cos(\omega t + \phi) - A_x \sin(\phi) \sin(\omega t + \phi) \quad (5)$$

Consider that the force $F_y(t)$ has two contributions: one is a restoring force, i.e. an elastic contribution F_{el} and the other is a damping force F_{damp} . The elastic force is directly proportional to the position of the moving mass, whereas the damping is directly proportional to the first derivative of the position i.e. to its velocity. Let us define k as being the proportionality constant between the force and the position and γ the proportionality constant between the damping force and the speed of the mass. Hence:

$$F_y(t) = F_{el}(t) + F_{damp}(t) = -kx(t) - \gamma \frac{dx(t)}{dt} \quad (6)$$

which means

$$F_y(t) = -kA \cos(\omega t + \phi) + \gamma\omega A \sin(\omega t + \phi) \quad (7)$$

comparing equation 7 with equation 5 gives:

$$\begin{aligned} -kA &= A_r - A_x \cos(\phi) \\ \gamma\omega A &= -A_x \sin(\phi) \end{aligned} \quad (8)$$

If the position $x(t)$ of the mass is known, then the total force $F_r(t)$ to which the moving mass is submitted to, (2) is also known:

$$\begin{aligned} k &= m\omega^2 + \frac{A_x}{A} \cos(\phi) \\ \gamma &= -\frac{A_x}{\omega A} \sin(\phi) \end{aligned} \quad (9)$$

Now suppose that the proportionality constant k between the force and the position depends on an external parameter z , suppose that it can be set $k = k_0 + k_i(z)$ and that in the same way it can be set $\gamma = \gamma_0 + \gamma_i(z)$. Then in such case:

$$\begin{aligned} k_i(z) &= -k_0 + m\omega^2 + \frac{A_x}{A} \cos(\phi) \\ \gamma_i(z) &= -\gamma_0 - \frac{A_x}{\omega A} \sin(\phi) \end{aligned} \quad (10)$$

Assume m , k_0 and γ_0 are all unknown constants. These constants may have effective values that depend on the frequency, hence we will consider solely the case where the frequency of motion ω is constant at all times, which is the common situation in AM-AFM. Suppose also to know $k_i(\infty) = 0$ and $\gamma_i(\infty) = 0$. Then, by setting the above equations equal to zero we obtain:

$$\begin{aligned} \frac{A_x}{A\omega} &= \sqrt{(k_0 - m\omega^2)^2 + \gamma_0^2 \omega^2} \equiv a \\ \phi^\infty &= \text{ArcTan} \left(\frac{\gamma_0 \omega}{k_0 - m\omega^2} \right) \end{aligned} \quad (11)$$

The superscript infinity means that those constants are evaluated far from the surface. In practice, due to the squeeze film effect, they should be evaluated before the short range forces but at few nanometers from that. We can then write a final relationship:

$$\begin{aligned} k_i(z) &= a[n \cos(\phi) - \cos(\phi^\infty)] \\ \gamma_i(z) &= \frac{a}{\omega} [\sin(\phi^\infty) - n \sin(\phi)] \end{aligned} \quad (12)$$

where we have put:

$$n \equiv \frac{A_x A_x^\infty}{A A_x^\infty} \quad (13)$$

The amplitudes of excitation and tip motion have been normalized to n which is then one far from the surface. As long as the excitation force A_x remains directly proportional to the excitation signal, it is not necessary to know its actual value, nor how is the mass actually excited. It does not matter if the mass is excited by displacing the base of the cantilever or through a direct actuation on the tip or a combination of both, as long as they remain proportional to the excitation signal. This formula is valid regardless of the presence of spurious peaks and or squeeze film effects, provided it can be assured the position is given in very good approximation by the expression in 1.

A question that could arise is whether there is any difference in having spurious peaks or not? $F_x(t)$ is the force acting on the moving mass. This force depends on the excitation signal that can be controlled. Spurious peaks will affect the ratio between F_x and the excitation signal as well as the phase between them. We will come back to this later on, and consider specifically one spurious peak. If the cantilever spectrum has well defined transfer function, then it is straightforward to obtain all constants above from that transfer function. If the spectrum however is deformed by spurious resonances, then that means that constants a and ϕ^∞ cannot be evaluated from a simple analysis of the spectrum. This does not mean however, that equations (12) are incorrect. An important note is that even if the resonance curve is calibrated close to the sample, we assume that k is the spring constant of the cantilever, so that a resonance frequency different from the natural frequency is accounted by only through a rescaling of the effective mass and quality factor. Whereas, if a and ϕ^∞ are calibrated, the cantilever spring constant is not fixed to any value.

The above assumption requires in practice that the interaction is linear. This can be assured by keeping the amplitude of oscillation small enough. It is assumed the measurement is the position of the particle to which the forces are applied. Most of the AFMs employ however optical beam deflection schemes [19], providing the measurement of the tip bending angle and not the tip position. If the cantilever is directly excited so that the base of the cantilever is not displaced [10, 11, 12, 13, 14], then equations (12) are still valid, because in that case the deflection

is indeed proportional to the position, implying that the measured signal is directly proportional to the total force applied on the tip. If the tip position is not measured and the cantilever is not directly excited, then equations (12) do not hold, particularly off the resonance or at the resonance but with small Q factor.

3 Coupling with one extra resonance

Consider now that the cantilever is coupled to another oscillator giving rise to one extra peak in the spectrum. The cantilever may be coupled in different ways. Let us consider firstly the case when the coupling is at the cantilever base, described by the equations:

$$\begin{aligned} m_l \ddot{x}_l + k_l(x_l - x_b) + \gamma_l \dot{x}_l &= 0 \quad (14) \\ m_s \ddot{x}_s + k_l(x_s - x_l) + k_s(x_s - x_0) + \gamma_s \dot{x}_s &= 0 \quad (15) \end{aligned}$$

This case corresponds to situations where additional resonances in the cantilever transfer function are due to the dither piezoelement in the cantilever holder or to the cantilever holder itself. Here the subscript l stands for lever and s for spurious. This system becomes straightforward to solve, considering spurious peaks at frequencies close to the cantilever frequency and assuming that nothing else in the system is as soft as the cantilever. If the frequency is comparable to the cantilever resonance frequency, then the ratio k_s/m_s is also comparable. If both the spurious motion is comparable to the cantilever motion, then the situation is such that the term $k_l(x_s - x_l)$ is negligible when compared to the other terms. This implies that the spurious motion is insensitive to motions of the cantilever i.e. the spurious motion is the same regardless of the cantilever vibrations. Thus, this motion depends only on the excitation signal and is the same regardless of the cantilever being present or not. As a matter of fact, if the set up cannot compel with this requirement, then the static spring constant of cantilever could not be used for any quantitative evaluation of the interaction, because in this case the deflection due to the tip-sample forces would not be directly proportional to the cantilever spring constant. As a consequence, the above condition/simplification corresponds to the real situation. By accepting this, equation (14) can be rewritten as:

$$\begin{aligned} m_l \ddot{x}_l + k_l(x_l - x_s) + \gamma_l \dot{x}_l &= 0 \\ m_s \ddot{x}_s + k_s(x_s - x_0) + \gamma_s \dot{x}_s &= 0 \end{aligned} \quad (16)$$

The second equation has a very well known steady state solution. A spurious oscillation will occur with amplitude: $A_s = x_0 / \sqrt{(k_s - m_s \omega^2)^2 + \gamma_s^2 \omega^2}$ and phase $\phi_s = \text{ArcTan}[\gamma_s \omega / (k_s - m_s \omega^2)]$. The problem then simplifies and summarizes in the solution of:

$$m_l \ddot{x}_l + k_l(x_l - A_s \cos(\omega t + \phi_s)) + \gamma_l \dot{x}_l = 0 \quad (17)$$

Which has the steady state solution given by:

$$A_l = \frac{k_l A_s}{\sqrt{(k_l - m\omega^2)^2 + \gamma_l^2 \omega^2}} \quad (18)$$

and phase respective to the excitation signal given by:

$$\phi_l = -\text{ArcTan}\left(\frac{\gamma_l \omega}{k_l - m_l \omega^2}\right) + \phi_s \quad (19)$$

Clearly, at a given frequency, the effect of a spurious resonance of the kind considered here is that the excitation signal is amplified by the spurious resonance (A_s) and a phase lag is introduced. However, if the frequency is kept constant, this amplification factor and phase lag remain constant. The quantity that matters $a = A_s/A_l$ remains unchanged. The effect of this spurious peak is that, depending on its resonance, the ratio between the actual excitation and the supplied excitation will change. We then conclude that this type of spurious peak do not affect the measured tip-sample interaction.

Consider now that the cantilever is coupled not at the base but at the tip position:

$$\begin{aligned} m_l \ddot{x}_l + k_l(x_l - x_b) + \gamma_l \dot{x}_l + k_c(x_l - x_c) &= 0 \\ m_c \ddot{x}_c + k_c(x_c - x_l) + \gamma_c \dot{x}_c + \dots &= 0 \end{aligned} \quad (20)$$

This case corresponds to a situation where additional resonances in the cantilever transfer function are due to the fluid borne excitation as described in reference [17]. Here, k_c represents a restoring force that may be induced by the fluid. Contrary to the previous case, now it is preferable to have a small value for constant k_c . We will not be able to solve the system above but we can rewrite the first equation as:

$$m_l \ddot{x}_l + (k_l + k_c)x_l + \gamma_l \dot{x}_l = x_b k_l + k_c x_c \quad (21)$$

Similarly to the previous case, both x_b and x_c are directly proportional to the excitation signal, so they add to give A_x also proportional to the excitation signal. We may not be able to tell how much is A_x . However, it is not difficult to see that in this case equation 12 still holds, except that now $k_0 = k_l + k_c$. Due to the coupling, the cantilever has a different effective and unknown spring constant. If constants a and ϕ^∞ are determined directly by using a known interaction, then the change in cantilever stiffness is directly taken into account. The constants a and ϕ^∞ can be calibrated by measuring tip position, oscillation amplitude and phase using a tip-sample known force or using for example a tip-sample approach curve and fitting $F = -\int k_i dz$ with equation 12 as in references [22] and [23]. The tip position provides the static force F , whereas amplitude and phase provide k_i through the calibrated constants a and ϕ^∞ . An electrostatic force may be easily employed in air for this purpose. In liquids it can be done for example by measuring tip-sample approach curves, assuming the static force to be conservative.

An intrinsic limit to any calibration method comes from the time dependence of the calibration parameters. If the

liquid cell has an open architecture, the evaporation of the liquid drop affects directly these parameters which are needed to quantify the interaction. They should then be evaluated continuously.

We employed the calibration method requiring the acquisition of tip-sample approach curves to compare measurements with a conventional acoustic excitation and a direct capacitive excitation of the tip. We coated an optic fiber, permitting the measurement of the tip position, with 30 nm thickness of gold at its end and 300 nm thickness of gold on its borders as shown in figure 1.

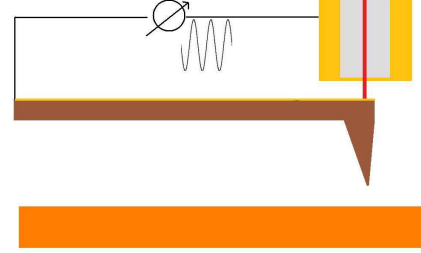


Figure 1: Setup employed for the direct excitation of the tip. A gold coated optic fiber is used to measure the tip position and to apply an electrostatic excitation of the conductive cantilever at a frequency close to resonance.

We applied a harmonic oscillating signal $V(\omega)$ around a given ΔV_0 . The force applied on the tip is given by:

$$F = -\frac{1}{2} \frac{\partial C}{\partial z} [\Delta V_0 + V(\omega)]^2 \quad (22)$$

where C is the capacitance resulting from the plates constituted by the cantilever backside and the bottom of the gold coated optical fiber. Figures 2a and b show the amplitude and the phase of the excited cantilever in air (red) and in deionized water (blue). Typical values for ΔV_0 are 5 V in air and 500 mV in liquid, whereas the amplitude of the harmonic modulation is 100 mV. The tip-fiber distance is 10 μm . At this distance, the force acting on 40 μm wide cantilevers is in the order of 10 nN/V in static conditions in air. In water no capacitive force acting on the cantilever was observed in quasi-static conditions, whereas figure 2a shows the presence of capacitive actuation at frequencies higher than 1 kHz. The diameter of the optic fiber is 125 μm . The cantilevers have nominal spring constant of 0.8 N/m.

Figures 2a and b show the amplitude and the phase of the cantilever in liquid with a direct excitation of the tip (blue) and with a conventional piezoelectric excitation (black), showing the presence of spurious resonances.

We acquired approach force curves in Force Feedback Mode [22, 23] at the mica/deionized water interface. The oscillation amplitude imposed to the tip is 0.3 nm. The amplitude and the phase were recorded and converted into conservative and dissipative interactions using equations 12 and the equality $F = -\int k_i dz$, assuming the static

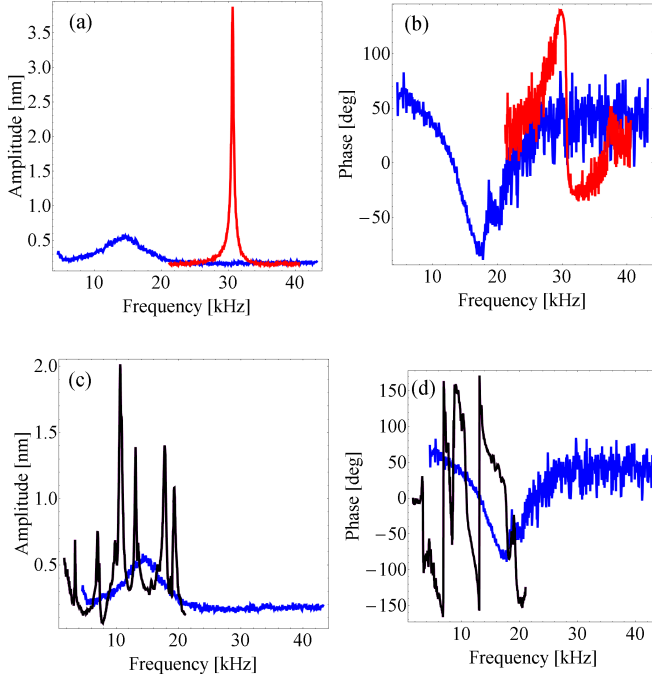


Figure 2: Excitation of the tip in air and in liquid with different actuation methods: a) Amplitude and b) phase of the tip directly excited in air (red) and (liquid). c) Amplitude and d) phase of the tip excited in liquid with a capacitive actuation (blue) and with a conventional piezoelectric excitation.

force to be fully conservative. Constants a and ϕ^∞ were determined. Then, we performed the same measurements exciting the tip with the same oscillation amplitude and with the conventional piezoelectric excitation. Even in presence of spurious peaks, we converted the amplitude and phase into conservative and dissipative interactions, providing new a and ϕ^∞ parameters. Figure 3a shows the static force measured while the tip was electrically excited (blue) and piezoelectric excited (red). The elastic force gradient and the dissipation are shown in figure 3b and 3c. Providing the same results in terms force gradient and dissipation, the main difference between the two excitation methods is then solely given by the calibration parameters a and ϕ^∞ evaluated through the equality $F = -\int k_i dz$.

In the case of electrostatic excitation, we obtained $\phi^\infty = 0.05$ rad, $a = 2.2$ N/m. For the conventional acoustic excitation, we have $\phi^\infty = 0.006$ rad, $a = 0.06$ N/m.

4 Measurement based on deflection angle

If the measurement is not directly proportional to the position of the cantilever but only proportional to its deflection, then the displacement of the base has to be added to the measured signal. We can start by considering

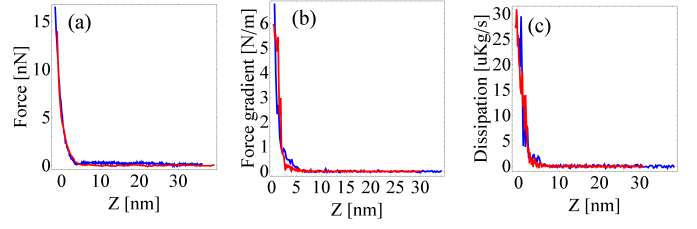


Figure 3: Interaction at the mica/deionized water interface with electrostatic excitation of the tip (blue) and conventional piezoelectric excitation (red): a) Static force. b) Force gradient. c) Dissipation.

equation 10 and simply recomputing both $\cos(\phi)/A$ and $\sin(\phi)/A$. The position of the moving mass is given by its deflection plus the motion of the base: $A \cos(\omega t + \phi) = A_m \cos(\omega t + \phi_m) + A_b \cos(\omega t + \phi_b)$. Where A_m is the amplitude of the measured signal and A_b the oscillation amplitude of the cantilever anchoring point. Equation 10 becomes:

$$\begin{aligned} k_i &= -k + m\omega^2 + \frac{A_x [A_m \cos(\phi_m) + A_b \cos(\phi_b)]}{A_m^2 + A_b^2 + 2A_b A_m \cos(\phi_m - \phi_b)} \\ \gamma_i &= -\gamma_0 - \frac{1}{\omega} \frac{A_x [A_m \sin(\phi_m) + A_b \sin(\phi_b)]}{A_m^2 + A_b^2 + 2A_b A_m \cos(\phi_m - \phi_b)} \end{aligned} \quad (23)$$

Note that equations 23 are exactly equal to equations 12 if one of the following is true:

1. The measured signal corresponds to the position of the moving tip, in which case the terms with A_b do not appear in the equation.
2. It is assured that the base of the cantilever does not move (direct excitation), in which case $A_b = 0$.
3. It is a very good approximation to state that $A_m \gg A_b$ (A_b is negligible compared to A_m). which is true close to the resonance frequency when the cantilever quality factor is large enough.

Interestingly, it means that to be able to use the equations described in the first part (12), it is convenient to either make a direct excitation of the cantilever or to measure directly a signal proportional to its position. In the last case, it becomes irrelevant how the moving mass is excited. There is one particular case worth noting when the excitation force is solely due to the cantilever base displacement. In this case it is $A_x = kA_b$ and $\phi_b = 0$.

Let us also define the proportionality constant r such that $nrA_m = A_x$. Here, r is a constant such that in the absence of tip-sample interactions the normalized amplitude (n) is one.

Equations 23 then become:

$$\begin{aligned} k_i &= k \left(\frac{m\omega^2}{k} - \frac{k(k + nr \cos(\phi_m))}{k^2 + n^2r^2 + 2knr \cos(\phi_m)} \right) \\ \gamma_i &= \frac{k}{\omega} \left(-\frac{\omega\gamma_0}{k} - \frac{knr \sin(\phi_m)}{k^2 + n^2r^2 + 2knr \cos(\phi_m)} \right) \end{aligned} \quad (24)$$

Far from the sample surface there are no short range sample interactions and equations (24) must evaluate to zero. We can solve equations (24) to find ϕ_m^∞ and r that are constants:

$$\begin{aligned} \frac{A_x^\infty}{A_m^\infty} &\equiv r = k \sqrt{\frac{\gamma^2\omega^2 + (k - m\omega^2)^2}{\gamma^2\omega^2 + m^2\omega^4}} \\ \phi_m^\infty &= \text{ArcCos} \left(\frac{\omega(\gamma^2 + m(m\omega^2 - k))}{\sqrt{(\gamma^2 + m^2\omega^2)(\gamma^2\omega^2 + (k - m\omega^2)^2)}} \right) \end{aligned} \quad (25)$$

Then, the tip-sample interaction is given by:

$$\begin{aligned} k_i &= k \left(\frac{k(k + r \cos(\phi_m^\infty))}{k^2 + r^2 + 2kr \cos(\phi_m^\infty)} - \frac{k(k + nr \cos(\phi_m))}{k^2 + n^2r^2 + 2knr \cos(\phi_m)} \right) \\ \gamma_i &= \frac{k}{\omega} \left(\frac{kr \sin(\phi_m^\infty)}{k^2 + r^2 + 2kr \cos(\phi_m^\infty)} - \frac{knr \sin(\phi_m)}{k^2 + n^2r^2 + 2knr \cos(\phi_m)} \right) \end{aligned} \quad (26)$$

This highlights the fact that if the cantilever spring constant k is known, then the evaluation of either γ and m or r and ϕ^∞ is needed to quantify the interaction. The first couple can be found through a resonance curve if it is well defined in the spectrum. In that case one can compute r from the expression above. The second couple can be found by taking a calibration curve using a known force or using for example an approach curve and fitting $F = -\int k_i dz$ as in [22,23]. In air it is straightforward to calibrate these parameters using an electrostatic force. If it is not possible to obtain these values from the resonance curve because the spectrum is very deformed, that does not mean these equations do not hold.

Finally, some considerations regarding the case where $A_x \neq kA_b$. First note that this situation poses no difficulty in the case where the tip motion is measured instead of the cantilever deflection. It is more complicated in the case of beam deflection because equation 23 is more difficult to analyze. The values of A_x and A_b , or their ratios, are not acquired from a conventional analysis of the transfer function nor from fitting the Brownian motion of the cantilever. Also note that this is exactly the case where piezoelectric actuation is used and liquid borne excitation is not negligible. However, one can use the equality $F = \int \nabla F dz$ to calibrate either equation 23 or 26 at a given frequency.

To illustrate the situation described above, we have prepared a cantilever holder that excites the cantilever by a combination of rotation and translation. In this case $A_x \neq kA_b$. Furthermore, this particular holder shows

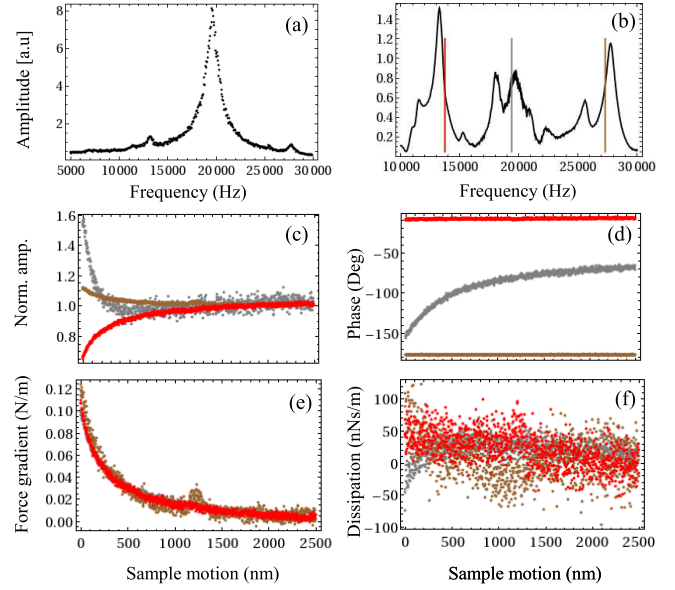


Figure 4: Characterization of a tip-sample electrostatic interaction at resonance (gray) and off resonance (red and brown) a) Cantilever Brownian motion; b) spectrum of the excited cantilever; c) normalized amplitude at three selected frequencies indicated in the spectrum; d) measured phase (offset so that at infinity it gives ϕ_m^∞); e) and f) force gradient and dissipation measured at the three selected frequencies.

many spurious resonances. Three approach curves were taken at selected arbitrary frequencies as shown in figure 4b. we applied an electrical potential difference between a conductive cantilever and a conductive sample which results in an electrostatic interaction. We have used equations 26 to account for the interaction. As shown in figure 4e, all the measurements provide the same force gradient regardless of the presence of spurious resonances shown in figure 4a, whereas the dissipation remains constant and approximately equal to zero. The static force was used to obtain all the unknown parameters. This result clearly shows that a calibration method is advantageous compared to a conventional analysis of the transfer function.

Interestingly, the values of r and ϕ^∞ that result from the calibration are those that would be expected if the spurious peaks were not there. Notice in figure 4d the phase close to 0 Deg, -90 Deg and -180 Deg, when the cantilever is respectively excited before, close to, and far from the resonance frequency. To ease the task of calibrating the cantilever, r and ϕ^∞ were initially guessed from equations 25, where the constants therein were obtained from the analysis of the tip Brownian motion shown in fig 4a. The resulting r^* and $\phi^{\infty*}$ are shown in table 1 and compared with r and ϕ^∞ obtained from the analysis of the Brownian motion. This result means that the change in amplitude and phase due to the interaction force were about the same, regardless of the presence of spurious peaks.

f_x	13734 Hz	19393 Hz	27315 Hz
r	1.05 N/m	0.059 N/m	0.48
r^*	1.05	0.056	0.4
ϕ^∞	-8 Deg	-66Deg	-178Deg
$\phi^{\infty*}$	-8 Deg	-66Deg	-177Deg

Table 1: Comparison between the calibration based on matching the integral of the force gradient to the force, and using directly the constants determined from fitting the Brownian motion of the tip. The * corresponds to values obtained from matching the integral of the force gradient to the force.

5 Conclusions

We have introduced a methodology to directly derive the conservative and dissipative interactions between the AFM probe and the sample in dynamic AFM experiments when small oscillation amplitudes of the tip are used and for different tip excitation and detection schemes. We considered both direct detection of the tip position (for example with Fabry-Perot interferometers) and classic optical beam deflection scheme, showing that the first method allows a much easier extraction of the conservative and the dissipative interactions without any knowledge of the effects of spurious resonances or the cantilever base motion. Even optical beam deflection based AFMs allows to extract the conservative and dissipative part of the interaction, performing the calibration procedure of the cantilever dynamics through the acquisition of tip-sample approach curves instead of the classic characterization through the cantilever transfer function.

Measuring directly the dip position greatly simplifies the analysis of the interaction. We have demonstrated in fact that the knowledge of the cantilever base motion is not necessary in order to quantify the conservative and dissipative interaction in dynamic AFM experiments in liquid if an interferometer is used to measure the tip position. The presence of spurious peaks related to the liquid cells resonances in the cantilever transfer function is known to introduce problems in the evaluation of the cantilever properties such as mass, spring constant and damping. We have proposed a calibration method of the cantilever response that is not based on the measurement of its transfer function, but it is based on the acquisition the tip position/deflection, amplitude and phase during approach curves and consequently the assumption of the equality $F = -\int k_z dz$. The method has been employed to compare the conservative and dissipative interaction at the mica/deionized water interface measured with a conventional piezoelectric dither excitation and direct electrostatic excitation. The different setups provide the same evaluation of the force gradient, although the calibration parameters are different. We have pointed out the limits of this method, which requires a continuous calibration of the cantilever parameters if the measurement is performed with a setup subject to evaporation of the liquid

drop. This limit is not intrinsic to the calibration method suggested in this work but is indeed common to any calibration procedure if the evaporation of the liquid drop is not avoided.

Finally, we have highlighted the changes that have to be introduced into the formulas for optical beam deflection based AFMs and we have shown how to quantify the conservative and dissipative part of a tip-sample electrostatic interaction in presence of spurious resonances in the cantilever transfer function.

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